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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ALUMINO SILICATE REFRACTORY MATERIALS

PART 9 DETERMINATION OF SODIUM AND POTASSIUM BY FLAME PHOTOMETRY

1. Scope — This standard (Part 9) covers a method for determination of sodium and potassium in alumino silicate refractory materials.

2. Determination of Sodium and Potassium by Flame Photometry

2.1 Outline of the Method — After fuming the sample to dryness with hydrofluoric acid and perchloric acid, the residue is dissolved in dilute hydrochloric acid and the solution is diluted to a definite volume. A portion of the sample is atomized in flame photometer. Concentration of alkalis present are computed by comparing the intensities of their radiations with those of the standard solution.

2.2 Reagents

- 2.2.1 Perchloric acid 70 percent.
- 2.2.2 Hydrofluoric acid 40 percent.
- **2.2.3** Dilute hydrochloric acid 1:1 (v/v).
- 2.2.4 Standard sodium solution Dissolve 0.254 2 g of dry sodium chloride in double distilled water and dilute to 500 ml in a volumetric flask (200 ppm Na). Prepare 1 to 15 ppm of solution by proper dilution.
- 2.2.5 Standard potassium solution Dissolve 0.190 7 g of dry potassium chloride in double distilled water and dilute to 500 ml in a volumetric flask (200 ppm Na). Prepare 1 to 15 ppm of solution by proper dilution.

2.3 Preparation of Sample Solution

- 2.3.1 Weigh 0.1 g of sample in a platinum basin, moisten with water and add to it, 1 ml of perchloric acid and 5 ml of hydrofluoric acid. Place the basin on a sand bath and evaporate till strong fumes of perchloric acid come out. Cool the basin and add 5 ml of hydrofluoric acid and repeat the process. Cool, wash the basin along the inner side with double distilled water and evaporate on sand bath to dryness.
- 2.3.2 To dry mass, add 5 drops of dilute hydrochloric acid and 20 ml of water. Place the basin on a steam bath. After 5-10 minutes, transfer quantitatively, the contents to a 250-ml volumetric flask and dilute to the mark. (If the solution is turbid, filter the solution through dry filter paper.)

2.4 Determination of Sodium

- 2.4.1 Place a few ml of solution in the cup of a flame photometer provided with monochromatic arrangement or sodium filter. Measure the emission at 589 nm or through appropriate filter against water.
- 2.4.2 Draw a calibration graph with 1-15 ppm concentration of standard sodium solutions. Calculate the unknown sodium concentration by referring to this graph.

2.5 Determination of Potassium

- 2.5.1 Place a few ml of the stock solution in the cup of a flame photometer. Measure the emission at 767 nm or through a potassium filter against water.
- 2.5.2 Draw a calibration graph with 1-15 concentrations of standard potassium solutions. Calculate the unknown potassium concentration by referring to this calibration graph.

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EXPLANATORY NOTE

Alumino silicate refractory materials contain alumina (Al_2O_3) and silica (SiO_2) in varying proportions made synthetically by heating aluminium trifluoride at 1 000-1 200°C with silica and water vapour.

It is used in kilns, laddles and furnaces that operate at higher temperatures or under conditions for which fire clay refractories are not suitable.

This Indian Standard has been prepared in several parts to cover the chemical analysis of various constituents in alumina silicate refractory materials. The other parts of the standard are:

- Part 1 Determination of loss on ignition
- Part 2 Determination of silica
- Part 3 Determination of aluminium
- Part 4 Determination of phosphorus
- Part 5 Determination of titanium
- Part 6 Determination of iron
- Part 7 Determination of menganese
- Part 8 Determination of calcium and magnesium